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(54) Title: METHOD FOR FABRICATING A PRODUCT COMPRISING SUBSTANTIALLY BIODEGRADABLE RAW MATERIALS

(57) Abstract

Method for fabricating a product comprising substantially biodegradable raw materials, a starting material being prepared from a base mixture containing biodegradable, natural materials or scraps and possibly binder and said starting material being subjected, in a die, to a pressure and heat treatment, characterized in that: a) a starting material is prepared which contains at least 8 wt.%, but no more than 18 wt.% of water, and b) the starting material obtained in step a) is moulded in the die at a temperature of at most 130 °C to give the product.

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Method for fabricating a product comprising substantially biodegradable raw materials

The invention relates to a method for fabricating a product comprising substantially biodegradable raw materials, a starting material being prepared from a base mixture containing substantially biodegradable, natural materials or scraps and binder and said starting material being subjected, in a die, to a pressure and heat treatment.

Such a method is known from the European Patent Application 613,905 by means of which a biodegradable product, in particular a biodegradable plant pot on the basis of natural vegetable or animal fibres is obtained which, based on the dry solids content, contains 50-90 wt% of fibres and 1-50 wt% of a pretreated potato pulp. According to this method, the potato pulp has to be pretreated, either with enzymes or mechanically, the potato pulp preferably being treated with enzymes and subjected, before and/or after said enzymatic treatment, to a mechanical pretreatment in the course of which potato pulp particles having a size of less than 350 µm are obtained. Said pretreatment is required in order to impart to the potato pulp particles binding properties with respect to the vegetable or animal fibres. The potato pulp which results from the production of starch and which contains about 75-95 wt% of water first of all has to be diluted to a solids content of less than 15 wt% and preferably less than 10 wt%. Then the potato pulp is treated with enzymes and/or is ground into small particles and is applied to the fibres, for example by impregnation or spraying. In a subsequent step, the excess water is removed, preferably mechanically, from the fibres impregnated or sprayed with potato pulp, after which a bonded fibre web is made under the influence of pressure and elevated temperature, and the product is moulded from said web. The product can also be fabricated by the pretreated potato pulp being processed, together with the vegetable or animal fibres, to produce a slurry, which slurry is then moulded with pressure and heat to give the product. The method according to the European Patent Application 613,905 has various drawbacks, however. For example, at least five to six steps are required (1. dilution and 2. enzymatic or mechanical pretreatment of the potato pulp, 3. application of the pretreated potato pulp to the fibres by impregnation or spraying, 4. removal of water, 5. making a bonded fibre web and 6. moulding the product or 1. dilution and 2. enzymatic or mechanical pretreatment of the potato pulp, 3. processing of the

pretreated potato pulp and the fibres to a slurry, 4. introducing the slurry into a press and 5. moulding the product under the influence of pressure and heat). Moreover, some of the steps are time-consuming: the treatment of the potato pulp with enzymes comprises a fermentation at 40°C for 20 hours. The method according to the European Patent Application 613,905 is therefore laborious and time-consuming.

Also known is a material which is marketed under the name "Fasal" and which is prepared from wood, maize, a binder and possibly other natural materials and inorganic environment-friendly colorants. The wood is used in the form of wood chips or sawdust and the maize in the form of cornflour or grits. The binder used is natural resin. The method for preparing the "Fasal" material comprises mixing the wood constituents and maize constituents in the dry state, after which the other constituents, some of which are in the liquid state, are added to this mixture to form a dispersible mixture. This mixture is then subjected to injection-moulding, a granular material being obtained in the process which consists of particles having a length of 3-5 mm and which has a moisture content of about 18%. Before this granular material can be used for the fabrication of a product, it first needs to be dried to a moisture content of about 10%. The method for fabricating products from the "Fasal" material therefore has the drawback that it is first necessary to produce a granular material and that this granular material then has to be dried to a suitable moisture content. A further drawback of this material is that if it used to fabricate a product, the thickness of the wall of the product has to be at least 3 mm. The mechanical properties of the "Fasal" material are therefore inadequate, in particular for fabricating products having a small wall thickness (for example 1-3 mm).

Other products comprising substantially biodegradable raw materials are described in the European Patent Application 716,804 and in the German Patent Application 19,500,653. According to the European Patent Application 716,804, biodegradable pots for seedlings can be fabricated by injection-moulding a mixture of coconut powder and a biodegradable plastic such as an aliphatic polyester which comprises a lactic acid resin and/or a carboxylic acid resin, the mixture containing 30-90 wt% of the resin and 70-10 wt% of coconut powder. The drawback of these pots is that an expensive plastic or a mixture of such plastics has to be used and that the pots are not completely degradable: after about 100 days, from about 5 to 20% of the pot still remains. The German Patent

Application 19,500,653 describes a container for plants which is formed by injection-moulding a mixture consisting of animal and/or vegetable fibres and fillers, pigments or other substances, together with a solution of alkali metal silicates in water. After the container has been moulded from this mixture, it is exposed to an acidic gas such as nitric acid or sulphuric acid, the alkali metal silicates decomposing to amorphous silicic acid and the container curing in the process. The container then needs to be conditioned by, for example, being dried. The method according to the German Patent Application 19,500,653 has the drawbacks that many steps are required for fabrication of the container (preparing mixture of the various constituents, moulding the product and treating it with an acidic gas, followed by conditioning) and that gaseous acids are required which are harmful to the environment and hazardous.

The object of the invention is to provide a solution to the abovementioned problems. The invention therefore relates to a method as mentioned in the preamble, in which:

a) a starting material is prepared which contains at least 8 wt%, but no more than 18 wt% of water, and

b) the starting material obtained in step a) is moulded in the die at a temperature of at most 130°C to give the product. It is therefore possible for the starting material not to contain any binder. Preferably, the starting material is prepared from from 70 to 99 wt% of vegetable materials or scraps and from 1 to 30 wt% of base mixture containing the binder.

The invention provides a method for a product which primarily or solely comprises biodegradable raw materials, the product being obtainable in a simple manner and in a short time. Moreover, no chemical substances are required which are harmful to the environment and hazardous, and the product degrades completely or almost completely over a specific time.

Preferably, in step a), the starting material is prepared from at least from 75 to 95 wt% of vegetable materials or scraps and from 5 to 25 wt% of base mixture containing the binder, in particular from from 80 to 90 wt% of vegetable materials or scraps and from 10 to 20 wt% of base mixture containing the binder, and in step b) the product is preferably moulded at a temperature of at most 120°C. The water which is present in the starting material derives from the water which was originally present in the original materials or scraps. It is therefore generally

unnecessary and often even undesirable to add additional water.

The starting material must not contain too little water, since that would make fabrication considerably more difficult, resulting in an unattractive product. Also, for the same reason, the temperature at which the product is moulded should not be too high. The starting material therefore preferably contains at least 10 wt% of water and in particular at least 12 wt% of water. The temperature at which the product is moulded is more preferably at most 110°C and in particular at most 100°C. If the product is moulded at a lower temperature, for example a temperature of less than 100°C, the starting material is preferably admixed with one or more viscosity-lowering preparations.

If the water content is too high, discoloration of the starting material may occur, which may lead to a product of unattractive appearance. It was also found that processing of a starting material having a high water content to obtain the product generally requires a lower temperature and low pressure. Since the temperature is not higher than 130°C, preferably at most 120°C, more preferably at most 110°C and in particular at most 100°C, the starting material therefore preferably contains not more than 16 wt% of water and in particular not more than 14 wt% of water.

The starting material is generally prepared from vegetable materials or scraps and a base mixture containing binder. According to a preferred embodiment, the binder is preferably a polysaccharide. The base mixture then preferably contains a release agent and a plasticizer. To obtain a product which can undergo biodegradation as rapidly and completely as possible, the starting material should contain as large an amount of vegetable materials or scraps as possible. The starting material can further contain natural colorants or natural pigments such as, for example, those used in foods. Examples of suitable colorants are chlorophyll, betanin, caramel, various carotenes and annatto.

The base mixture containing binder preferably contains a release agent and a plasticizer, the binder ensuring that a product having sufficient strength is obtained, the release agent ensuring that the product, once it has been moulded, can be removed from the die without difficulty, and the plasticizer being required for effective action of the binder. Based on the binder, the base mixture contains from 1 to 5 wt% of the release agent, preferably from 1.5 to 4 wt% and in particular from 2 to 3 wt%, and from 5 to 30 wt% of the plasticizer, preferably from 8 to 25 wt% and in particular from 10 to 20 wt%. Examples

of suitable binders are polysaccharides such as, for example, starch-type, pectin-type, cellulose-type substances and customary plastics. Examples of suitable release agents are phospholipids such as lecithins. Examples of suitable plasticizers are polar solvents such as water, aliphatic alcohols, diols or triols such as ethanol, glycol, 1,3-dihydroxypropane, glycerol and higher aliphatic alcohols such as pentaerythritol, di- and triethylene glycols and di- and tripropylene glycols and alkyl ether derivatives, for example methyl ether derivatives, and acetate derivatives thereof. The binder is preferably a starch and in particular potato starch, a cold-solvent-soluble starch, for example "Flocgel", or a modified starch. The release agent is preferably a lecithin and in particular lecithin obtained from soya. The polar solvent is preferably an aliphatic triol and in particular glycerol, natural glycerol being used in preference to synthetic glycerol. The base mixture may also contain a preparation for increasing the density of the base mixture, such as lignosulphate.

The starting material prepared from vegetable materials or scraps generally comprises vegetable materials or scraps such as scraps of wood, vegetable and garden waste, dried herbs, dried vegetables or scraps thereof, dried flowers or remains thereof, dried fruit or remains thereof, scraps of paper, cotton fibres, hay, straw, dried leaves, flax, hemp, elephant grass or mixtures thereof. Examples of suitable dried herbs are chervil and stalks thereof, parsley and turmeric. Examples of suitable vegetables or scraps thereof are carrots, beet and cabbage. Examples of suitable flowers or remains thereof are tulips, roses, hibiscus, cornflowers, marigolds and lavender. Suitable examples of dried leaves are those from birch and stinging-nettle. By using vegetable materials or scraps which are coloured, and by optionally using natural colorants, it is possible to obtain a product having an attractive appearance.

It was found that the product can be fabricated from a starting material entirely consisting of vegetable materials or scraps, if these materials or scraps are derived from carrots, green beans or peas.

The starting material preferably comprises scraps of wood, in particular chips and sawdust, and other biodegradable vegetable materials or scraps such as the abovementioned, preferably dried vegetable and garden waste, dried herbs, dried vegetables or scraps thereof, dried flowers or remains thereof, dried fruit or remains thereof, paper scraps, cotton fibres, hay, straw, dried leaves or mixtures thereof. The starting

material generally contains from 1 to 99 wt%, preferably from 5 to 90 wt% and in particular from 10 to 95 wt% of scraps of wood.

The starting material is generally in fibrous and/or particulate form and is obtained by mixing of the desired constituents. The fibres or particles generally have a size of from 0.01 to 10 mm, preferably from 0.1 to 5 mm and in particular from 1 to 3 mm. The starting material can be processed directly to give the product according to the invention. It is possible, however, for the starting material first to be pelleted or extruded, granules having a size of from about 1 to 20 mm, preferably having a size of from 1 to 5 mm being obtained in the process.

The product according to the invention is then moulded by means of compression- or injection-moulding, the starting material being processed directly as such, i.e. in its fibrous and particulate form, or the granules comprising the starting material being processed. The compression- or injection-moulding involves the use of a die whose shape is complementary to that of the desired product. Thus products can be moulded which have sufficient strength, and generally have a wall thickness of at least 1 mm. Thus products can be fabricated having a wall thickness of 1-3 mm. The upper limit of the wall thickness is in fact not limited and can, for example, be 1-5 cm. It will be obvious to those skilled in the art that the method according to the present invention can also be used to fabricate solid products.

If the product is formed by compression-moulding, the starting material can contain much more than 30 wt% of the base mixture containing binder, for example 70 wt%. According to the invention, the product can therefore be fabricated from a starting material, said starting material being compression-moulded at a temperature of at most 130°C. According to the invention, said product is preferably fabricated from a starting material which contains at least from 50 to 100 wt%, more preferably from 60 to 99 wt% and in particular from 75 to 95 wt% of vegetable materials or scraps and from 0 to 50 wt%, more preferably from 1 to 40 wt% and in particular from 5 to 25 wt% of base mixture containing a binder.

The starting material according to the invention can be used to mould products which have excellent biodegradability. The starting material according to the invention is suitable, in particular, for moulding plant pots and trays, coffins, toys, disposable goods such as hard packaging, gift articles, office articles, body-care and cosmetic products, motor car articles, decorative products, domestic products and

buffer materials. The invention therefore also relates to a product obtainable in accordance with the method as described. The product according to the invention may, if required, be made water-repellent by being coated with, for example, a water-insoluble protein or with a biodegradable, preferably natural wax such as beeswax. Making the product water-repellent can be effected by the base mixture being admixed with a biodegradable polymer such as poly(lactic acid), polycaprolactone, butene-succinate-adipate copolymers and starch-polyester blends.

The invention will be explained in more detail with reference to a few examples.

Example 1

A base mixture was prepared by mixing of potato starch, lecithin obtained from soya, and natural glycerol. The properties of the lecithin and the glycerol are shown in the tables below. The base mixture contained 2 wt% of lecithin, 14 wt% of glycerol and 84 wt% of potato starch.

Lecithin

Phospholipid (as acetone-insoluble)	min. 62%
Water	max. 0.8%
Acid value	max. 30
Peroxide value	max. 3
Iodine value (in 10%)	max. 60
Suspended matter (as toluene-insoluble)	max. 0.20%
Viscosity (mPa.s; 25°C)	12,000
Total microbial count/g	max. 3000

Glycerol

Purity	min. 99.7 wt%
Density (20°C)	1.263 kg/l
Colour Pt-Co scale	max. 10
pH	about 7
Viscosity	about 1300 mPa.s
Water content	max. 0.2 wt%
Saponification value	max. 0.64 meq/100 g

3 kg of this base mixture were admixed with 7 kg of vegetable material, said material consisting of 70 wt% of wood fibres and 30 wt% of chervil stalks.

Example 2

The starting material of Example 1 was injection-moulded with the aid of an injection-moulding machine of type DEMAG 25 (the screw diameter was 18 mm) to produce rods, with the proviso that the starting material had been prepared from a base mixture containing 30% of potato starch and that the starting material, prior to injection-moulding, was pelleted to give granules having a diameter of about 5 mm and a length of about 12 mm. The water content of the starting material was 12.5%.

The injection-moulding conditions were as follows:

Die temperature	30°C
Cylinder temperature (trial 1)	35/110/115/120/120°C
Cylinder temperature (trial 2)	35/95/95/100/100°C
Injection pressure	max. 2400 bar
Speed	200 rpm
After-pressure time	about 10 s
After-pressure	500 bar
Injection time	< 1 s
Cycle time	about 50 s

In trial 1, browning of the rods was observed, which in trial 2 occurred to only a very limited extent.

If the trial according to this example was repeated with a starting material which consisted of 2 kg of the base material according

to Example 1 and 8 kg of vegetable material according to Example 1, a comparable result was achieved.

Example 3

A starting material was prepared from 70 wt% of sawdust and 30 wt% of a base mixture which contained 84 wt% of starch, 2 wt% of lecithin and 14 wt% of glycerol. The starting material was made into pellets and these were then compression-moulded, with the aid of a die, to give a round bowl-shaped product having a diameter of 41 mm, a height of 28 mm and a wall thickness of 1.5 mm. The pressing time was 3 sec, and compression-moulding was carried out with a force of 80 metric tonnes and a die temperature of 70°C.

CLAIMS

1. Method for fabricating a product comprising substantially biodegradable raw materials, a starting material being prepared from a base mixture containing biodegradable, natural materials or scraps and possibly binder and said starting material being subjected, in a die, to a pressure and heat treatment, characterized in that:

a) a starting material is prepared which contains at least 8 wt%, but no more than 18 wt% of water, and

b) the starting material obtained in step a) is moulded in the die at a temperature of at most 130°C to give the product.

2. Method according to Claim 1, characterized in that the starting material is prepared from from 70 to 99 wt% of vegetable materials or scraps and from 1 to 30 wt% of base mixture containing the binder.

3. Method according to Claim 1 or Claim 2, characterized in that the starting material contains from 75 to 95 wt% of vegetable materials or scraps.

4. Method according to any one of the preceding claims, characterized in that the product is moulded at a temperature of at most 120°C.

5. Method according to any one of the preceding claims, characterized in that the binder is a polysaccharide.

6. Method according to Claim 5, characterized in that the base mixture containing binder contains at least one release agent and a plasticizer.

7. Method according to Claim 5 or Claim 6, characterized in that, based on the binder, the base mixture containing binder contains from 1 to 5 wt% of the release agent and from 10 to 30 wt% of the plasticizer.

8. Method according to any one of Claims 5-7, characterized in that the release agent is a lecithin and the plasticizer is a polar solvent.

9. Method according to any one of the preceding claims, characterized in that the vegetable materials or scraps are scraps of wood, dried herbs, dried vegetables or scraps thereof, dried flowers or remains thereof, dried fruit or remains thereof, scraps of paper, cotton fibres, hay, straw, dried leaves or mixtures thereof.

10. Method according to any one of the preceding claims, characterized in that the vegetable materials or scraps contain particles of wood having a size of from 0.01 to 10 mm.

11. Method according to any one of the preceding claims, characterized in that the starting material, prior to step b) is pelleted or extruded to produce particles having a diameter of from 1 to 20 mm.

12. Method according to any one of the preceding claims, characterized in that the product is moulded by means of compression- or injection-moulding.

13. Method according to any one of the preceding claims, characterized in that the product is moulded by means of compression-moulding.

14. Method according to any one of the preceding claims, characterized in that the starting material is prepared from from 50 to 100 wt% of vegetable materials or scraps and from 0 to 50 wt% of base mixture containing the binder.

15. Product obtainable in accordance with the method according to one or more of Claims 1 to 14.

16. Product according to Claim 15, characterized in that the product has a wall thickness of at least 1 mm.

AMENDED CLAIMS

[received by the International Bureau on 15 June 1998 (15.06.98); original claims 1, 2, 6, 7 and 14 amended; remaining claims unchanged (2 pages)]

1. Method for fabricating a product comprising substantially biodegradable raw materials, a starting material being prepared from biodegradable, natural materials or scraps and optionally a binder containing base mixture and said starting material being subjected, in a die, to a pressure and heat treatment, characterized in that:

a) a starting material is prepared which contains at least 8 wt%, but no more than 18 wt% of water, and

b) the starting material obtained in step a) is moulded in the die as such or in the form of particles at a temperature of at most 130°C to give the product.

2. Method according to Claim 1, characterized in that the starting material is prepared from 70 to 99 wt% of vegetable materials or scraps and from 1 to 30 wt% of the binder containing base mixture.

3. Method according to Claim 1 or Claim 2, characterized in that the starting material contains from 75 to 95 wt% of vegetable materials or scraps.

4. Method according to any one of the preceding claims, characterized in that the product is moulded at a temperature of at most 120°C.

5. Method according to any one of the preceding claims, characterized in that the binder is a polysaccharide.

6. Method according to Claim 5, characterized in that the binder containing base mixture contains at least one release agent and a plasticizer.

7. Method according to Claim 5 or Claim 6, characterized in that, based on the binder, the binder containing base mixture contains from 1 to 5 wt% of the release agent and from 10 to 30 wt% of the plasticizer.

8. Method according to any one of Claims 5-7, characterized in that the release agent is a lecithin and the plasticizer is a polar solvent.

9. Method according to any one of the preceding claims, characterized in that the vegetable materials or scraps are scraps of wood, dried herbs, dried vegetables or scraps thereof, dried flowers or remains thereof, dried fruit or remains thereof, scraps of paper, cotton fibres, hay, straw, dried leaves or mixtures thereof.

10. Method according to any one of the preceding claims, characterized in that the vegetable materials or scraps contain particles of wood having a size of from 0.01 to 10 mm.

11. Method according to any one of the preceding claims, characterized in that the starting material, prior to step b) is pelleted or extruded to produce particles having a diameter of from 1 to 20 mm.

12. Method according to any one of the preceding claims, characterized in that the product is moulded by means of compression- or injection-moulding.

13. Method according to any one of the preceding claims, characterized in that the product is moulded by means of compression-moulding.

14. Method according to any one of the preceding claims, characterized in that the starting material is prepared from from 50 to 100 wt% of vegetable materials or scraps and from 0 to 50 wt% of the binder containing base mixture.

15. Product obtainable in accordance with the method according to one or more of Claims 1 to 14.

16. Product according to Claim 15, characterized in that the product has a wall thickness of at least 1 mm.

Statement under Article 19 PCT

EP-A-0.753.541 discloses that the fibers from which the product is manufactured are loosened. According to the present application the product is manufactured from the starting material as such or in the form of particles.

INTERNATIONAL SEARCH REPORT

Int. ional Application No
PCT/NL 98/00009

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 A01G9/10

According to International Patent Classification(IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 A01G C08L

Documentation searched other than minimumdocumentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 95 04111 A (RETTENBACHER ET AL.) 9 February 1995 see the whole document	1,4,5,9, 10,12, 13,15
A	---	2,3,6, 11,14,16
X,P	EP 0 753 541 A (BCT AG) 15 January 1997 see the whole document	1-4,9, 10,12-16
A	---	
	EP 0 613 905 A (WIBMER & CO.) 7 September 1994 cited in the application see the whole document	1-4,9, 10,14-16

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of theinternational search	Date of mailing of the international search report
1 April 1998	14/04/1998

Name and mailing address of the ISA	Authorized officer
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INTERNATIONAL SEARCH REPORT

Int ional Application No
PCT/NL 98/00009

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
A	WO 96 18675 A (CO-PAK) 20 June 1996 see the whole document	1-4,9, 11-13,15
A	EP 0 716 804 A (SUGIMOTO) 19 June 1996 cited in the application see the whole document	1-3,9, 11,12, 14-16
A	EP 0 559 450 A (HAYASHIBARA SEIBUTSU KAGAKU KENKYUJO) 8 September 1993 see page 1, line 48 - page 2, line 56; examples B-7	1,4,5, 12,15
A	DE 195 00 653 A (NÜRNBERGER) 18 July 1996 cited in the application see claims	1,4,9, 10,14,15

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Form PCT/ISA/210 (continuation of second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

Information on patent family members

In ıtional Application No
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